

A robust method to acquire tilt series in a few seconds for Fast Operando Nano-Tomography in ETEM

Grenier, T.¹, Roiban, L.², Banjak, H.¹, Koneti, S.², Maxim, V.¹ and Epicier, T.²

¹ Univ. Lyon, CREATIS, umr 5510 CNRS, INSERM U1206, INSA-Lyon, University Claude Bernard Lyon 1, France, ² Univ. Lyon, MATEIS, umr 5510 CNRS, INSA-Lyon, University Claude Bernard Lyon 1, France

The constant development of operando Transmission Electron Microscope (TEM) such as in Environmental TEM (ETEM) raises naturally the question of 3D analyses during a dynamic evolution of nanomaterials. Obviously, the required series of projections for tilting tomography must be acquired very quickly to allow pertinent volumes reconstruction of a sample exhibiting fast shape or structure changes when exposed to external stimuli (temperature and gas in ETEM). Using tomographic holders in modern instruments coupled with high speed cameras operated in the video mode at several hundreds of frames per second (fps) allows tilting series to be acquired in a few seconds during a fast continuous rotation of the sample [1,2]. We use a FEI-Titan ETEM equipped with a Gatan OneView camera and a DENS Solutions Wildfire heating holder to develop Fast Operando Nano Tomography (as introduced in [3]). A key issue limiting its efficiency is the speed, reproducibility and robustness of drift corrections over a large tilt amplitude (e.g. 140°). It is impossible to correct directly in a few seconds the lateral displacements of the projected object which frequently exits the camera field of view even if the eucentric position is properly adjusted. We develop here an accurate and robust 'fastomo' method to overcome this problem (Figure 1). The projected trajectory of the sample is first pre-calibrated during a 'test' tilt series, from which drift corrections that appeared to be reproducible during consecutive tilt operations are deduced. This tracking procedure, including decoding the DM4 in situ image sequence, takes only 2 to 6 seconds using our in-house optimized software based on a Normed Cross-Correlation in Fourier Space (NCCFS) [4, 5]. This speed is crucial because any sample is permanently evolving under operando conditions in ETEM. Drift corrections that need to be measured only once in a given area are then automatically and regularly applied during the continuous recording of the 'true' tilt series without perturbing the acquisition. Fast 3D ETEM studies of nanocatalysts will be illustrated [6].

[1] V. Migunov et al., Sci. Reports, 5 (2015), 14516

[2] L. Roiban et al., Microsc. Microanal. 22, Suppl 5 (2016), 8

[3] L. Roiban et al., J. of Microscopy, 269, 2 (2018), 117

[4] J.P. Lewis, Vision Interface, 10 (1995) 120-123

[5] G. Bradski, Dr. Dobb's Journal, November 01 (2000); also <http://github.com/itseez/opencv>

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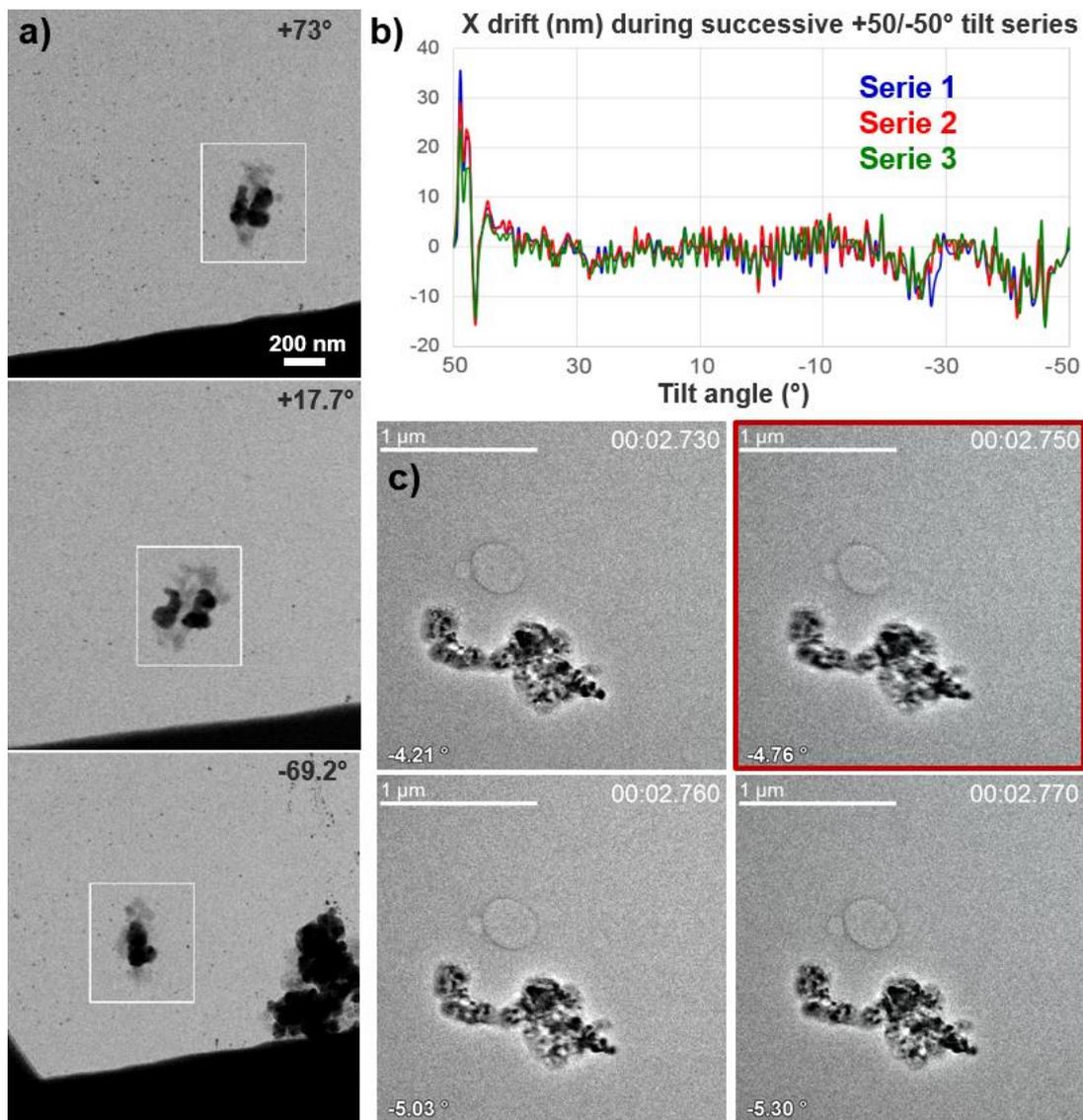


Figure 1: The 'fastomo' approach. a): successful NCCFS-based fast tracking of a soot-ZrO₂ aggregate (white frame) during a +73 to -70° tilt series (1Kx1K images) recorded in 5.1 s at 100 fps in ETEM (300°C): measurements are performed every 30 frames (3 frames shown, 4 s tracking procedure). b): Reproducible drifts during 3 successive tilt series measured as shown in a). c): 5 s +70 to -70° tilt series of CeO₂ particles: 4 successive frames (2Kx2K) are shown when applying a drift correction (red frame).